

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: )  
Hisashi ICHIMURA et al. )  
Serial No.: 09/902,256 )  
Filed: July 11, 2001 ) Group Art Unit:1751  
ANIMAL FIBER SUPERIOR IN ) Examiner: Preeti Kumar  
SHRINK PROOFING AND )  
METHOD FOR PREPARATION )  
THEREOF )

DECLARATION PURSUANT TO 37 C.F.R. §1.132

Honorable Commissioner of Patents and Trademarks  
Washington, D.C. 20231

Sir:

I, Kazuhiro Nakase, declare that:

1. I am one of the co-inventors of the invention disclosed and claimed in the application identified in caption.

2. I currently reside at 1-46-1-407 Kisabenishi, Katano, Osaka 572-0041 Japan.

3. I received the degree of Master of Applied Reaction Chemistry from Graduate School of Engineering Department of FUKUI University on March 31, 1992.

4. I have been employed since April 1, 1992 by KURABO INDUSTRIES LTD., the assignee of the present application, and have been engaged in research and development of fiber processing technology (including wool fiber) for recent 4 years.

5. I conducted the experiments in order to demonstrate the difference of modified wools treated according to Hojo (United States Patent No. 5,824,113) and the present invention, and I beg to submit an exact report thereon.

6. Experiment

### ① Samples

#### (i) Sample 1: Non-treated wool sliver

Wool sliver (of 25 g/m of grain and, 75.0mm of average fiber length) made of Merino wool of 20.7  $\mu$  from Australia was used as Sample 1.

(ii) Sample 2: Wool sliver of Sample 1 was treated in the same manner as Example 1 and Example 3 of the present application to give Sample 2-1 (according to Example 1) and Sample 2-2 (according to Example 3).

(iii) Sample 3: Wool sliver of Sample 1 was treated in the same manner as Example 1 of Hojo (USP No 5,824,113) to give Sample 3 (according to Hojo).

### ② Evaluation and Result

Evaluation was made with respect to (i) Observation of the surface of the Scales of the Sample Wool, (ii) FT-IR measurement of the Sample Wool, (iii) Water Repellency of Sample Wool (Sink-Float Method), (iv) Allwörden reaction (Confirmation of existence of epicuticle layer), (v) Felting Shrinkage Percentage and (vi) Pilling Resistance as below. The results are summarized in Table 1 below.

#### (i) Observation of Scales

Pt-Pd was deposited on the Wool fibers of each Sample 1 to 3 (10 nm thickness) by Ion-Sputtering Apparatus E-1030 (made by Hitachi, Ltd.).

The surface of the wool fiber was observed (with magnification of 1,500) by Scanning Electron Microscope S-4500N (made by Hitachi, Ltd.) to observe whether scales existed on the surface of wool of Samples or not.

Scales were not observed with respect to Sample 3 (Hojo)

as shown in Fig. 1(A)-Fig. 1(D).

This shows that Sample 3 (Hojo) has a completely different form from original wool surface form possessed by Sample 1 (non treated original wool), Samples 2-1 and 2-2 of the present invention.

(ii) FT-IR measurement

Wool fibers of each Sample 1 to 3 were subjected to reflection FT-IR measurement (ATR method) using Fourier Transform Infrared Spectroscope Nicolet Magna 760 type (made by Nicolet) to give relative absorbance in the absorption band of  $\text{-SO}_3\text{H}$  group ( $1040\text{ cm}^{-1}$ ) and  $\text{-S-SO}_3\text{Na}$  group (Bunte salts) ( $1024\text{ cm}^{-1}$ ) with the absorbance of the absorption band corresponding to amide I set to 1.

Relative absorbance in the absorption band of  $\text{-S-SO}_3\text{Na}$  group (Bunte salts) ( $1024\text{ cm}^{-1}$ ) is zero with respect to Sample 1 (non-treated) and Sample 3 (Hojo) as shown in Fig. 2 and Fig. 3. This shows that Samples 2-1 and 2-2 has completely different structure from that of Sample 1 (non-treated) or Sample 3 (Hojo).

This enables to differentiate of the wool of the present invention from Hojo.

(iii) Water Repellency (Shink-Float Method)

For measuring water repellency of each Sample Wool, the sliver was fiber-opened, and 1 g was sampled, 800 mL of distilled water was charged into a 1-L beaker and each Sample wool was floated on the water surface and sedimentation condition was observed.

Sample 3 (Hojo) did not have water repellency, as shown in Fig. 4. This shows that Sample 3 (Hojo) does not have

epicuticle layer which is originally possessed by wool as possessed by Sample 1 (non-treated) and Samples 2-1 and 2-2 of the present invention.

This enables to differentiate of the wool of the present invention from Hojo.

(iv) Allwörden reaction

Several wool single fibers of each Sample were put on a glass plate, and several droplets of saturated bromine water were dropped thereon, and immediately after this, the state of the surface of each fiber was observed under an optical microscope (Nikon OPTIPHOT) (magnification of 200). When any epicuticle layer in scales existed, bubbles generated on the surface of the fiber. Therefore, the existence of any epicuticle layer in scales was confirmed depending on the generation of bubbles.

Sample 3 (Hojo) did not generate bubbles while Samples 1, 2-1 and 2-2 generated bubbles, as shown in Fig. 5(A) to 5(D). It was confirmed by the result that while Samples 1, 2-1 and 2-2 have epicuticle layer as possessed by the original wool, Sample 3 does not have epicuticle layer.

This enables to differentiate the wool of the present invention from Hojo.

(v) Felting Shrinkage Percentage

2/48Nm worsted yarn was spun at twist number of Z480 × S270 to be knitted into a cover-factor C.F. 0.41 with one thread being taken from 14 gages to give a knitted fabric specified in accordance with testing procedure of Wool Mark Test Method 31.

Each knitted fabric formed of each Sample was measured

with respect to Felting Shrinkage Percentage in conformity with WM TM31 method (Wool Mark Test Method 31), while the test washing machine was changed to a Cubex shrinkage testing machine and the washing time was 3 continuous hours.

Sample 1 (non-treated) and Sample 3 (Hojo) had Felting Shrinkage Percentage of 72.1 % and 38.4 % respectively.

This enables to differentiate of the wool of the present invention from Hojo.

(vi) Pilling Resistance

2/48Nm worsted yarn was spun at twist number of 2480 × S270 to be knitted into a cover-factor C.F. 0.48 with one thread being taken from 18 gage to give a knitted fabric.

Each fabric formed of each Sample was evaluated with respect to piling resistance in accordance with JIS L 1076 6.1A using ICI-type pill box test machine.

Sample 1 (non-treated) and Sample 3 (Hojo) had 1-2 class.

This enables to differentiate of the wool of the present invention from Hojo.

Table 1

		Sample 1 (not-treated)	Sample 2-1 (Example 1)	Sample 2-2 (Example 3)	Sample 3 (Hojo)
Observation by Electron Microscope	Existence or Non-Existence of Scales	Scales (Fig.1(A))	Scales (Fig.1(B))	Scales (Fig.1(C))	No-Scales (Fig.1(D))
FT-IR (Fig.2, Fig.3)	-SO <sub>3</sub> -H(1040cm <sup>-1</sup> )	0.06	0.14	0.16	0.19
	-S-SO <sub>3</sub> Na(1024cm <sup>-1</sup> )	0	0.29	0.28	0
Water- Repellency	Sedimentation (Fig.4)	No Sedimentation (Fig.4)	No Sedimentation (Fig.4)	No Sedimentation (Fig.4)	Instant Sedimentation (Fig.4)
Allwörden Reaction	Generation of Bubbles (Confirmation of Epicuticle Layer)	Generation of Bubbles (Existence of Epicuticle Layer)Fig.5(A)	Generation of Bubbles (Existence of Epicuticle Layer)Fig.5(B)	Generation of Bubbles (Existence of Epicuticle Layer)Fig.5(C)	No Generation of Bubble (Non- Existence of Epicuticle Layer)Fig.5(D)
Felt Shrinkage Rate (% area)		-72.1 <sup>*1)</sup>	-1.0	-3.9	-38.4
Pilling Resistant (Class)		1-2	4	3-4	1-2

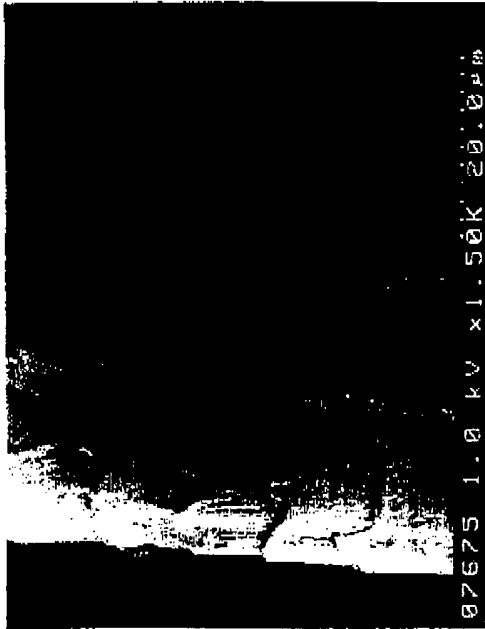
\*1) Minus means shrinkage

7. It is declared by the undersigned that all statements made herein of undersigned's own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonments, or both, under 18 U.S. Code 1001 and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

Dated this 6<sup>th</sup> day of October, 2003

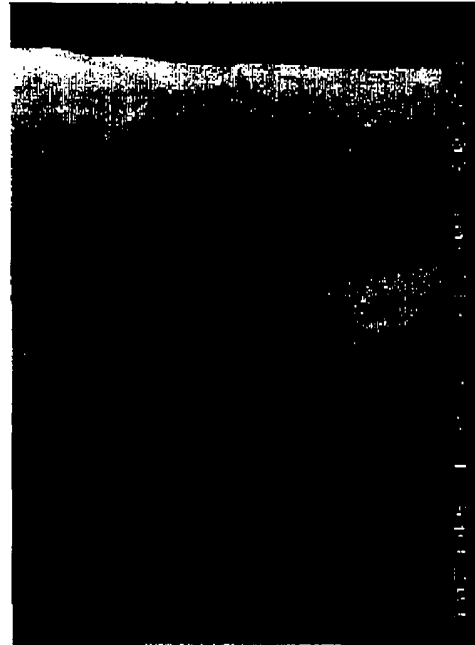
*Kazuhiro Nakase*  
Kazuhiro Nakase

Fig. 1(B)



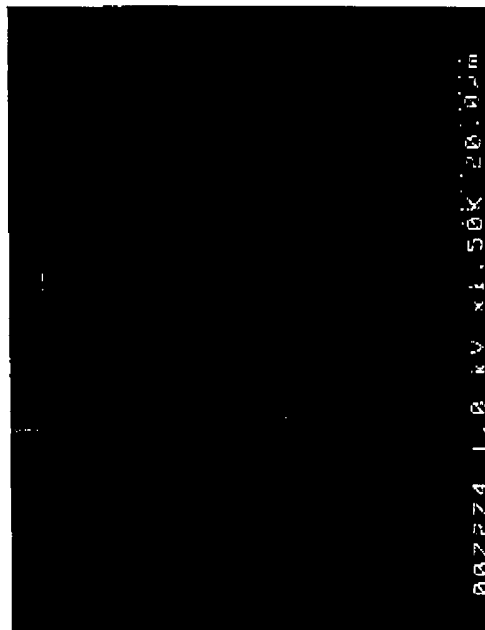
Sample 2-1

Fig. 1(D)



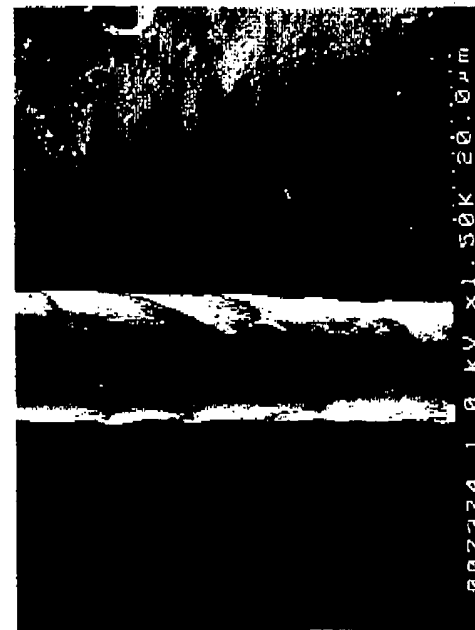
Sample 3

Fig. 1(A)



Sample 1

Fig. 1(C)



Sample 2-2

Fig. 2

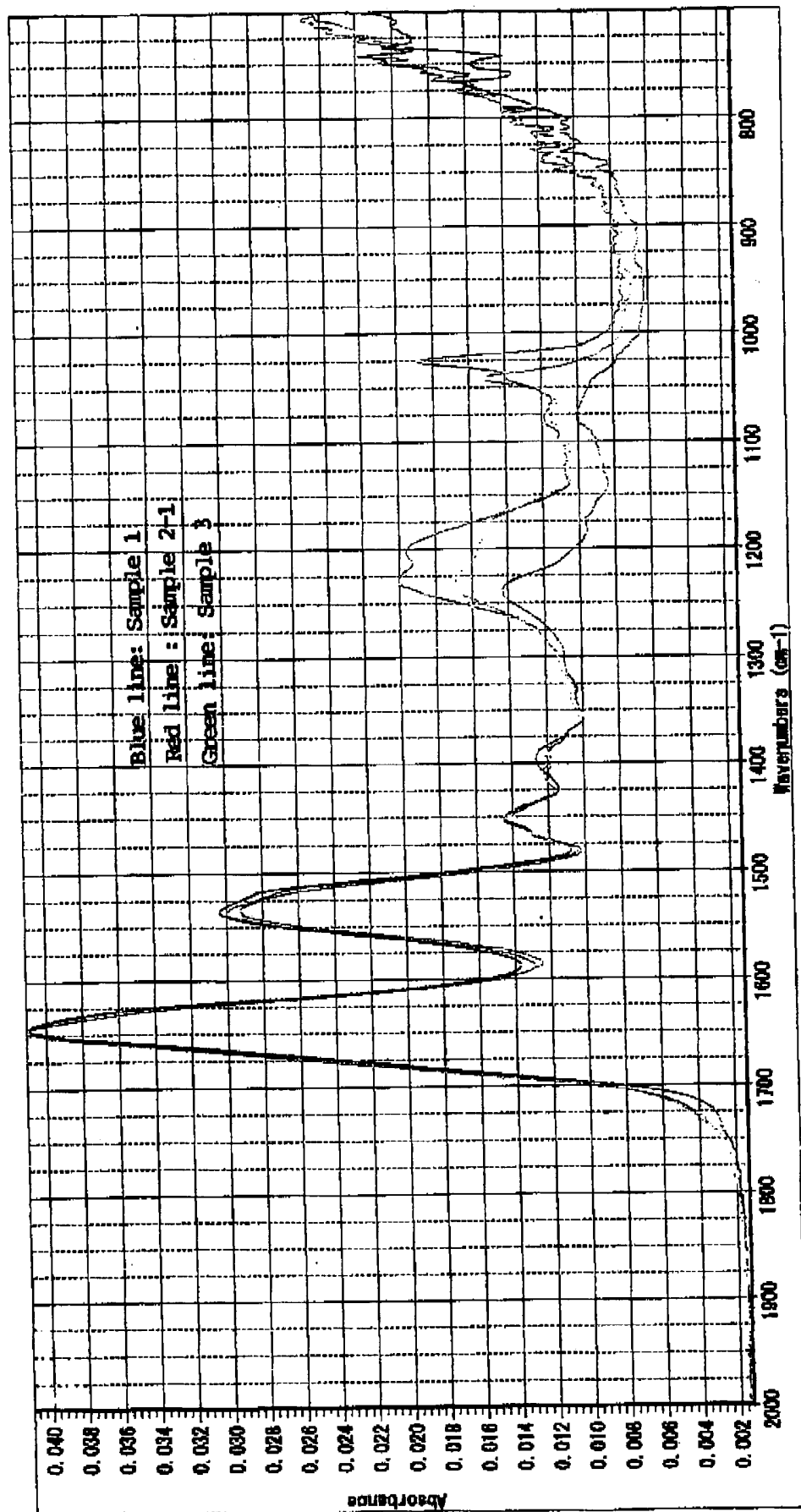




Fig. 3

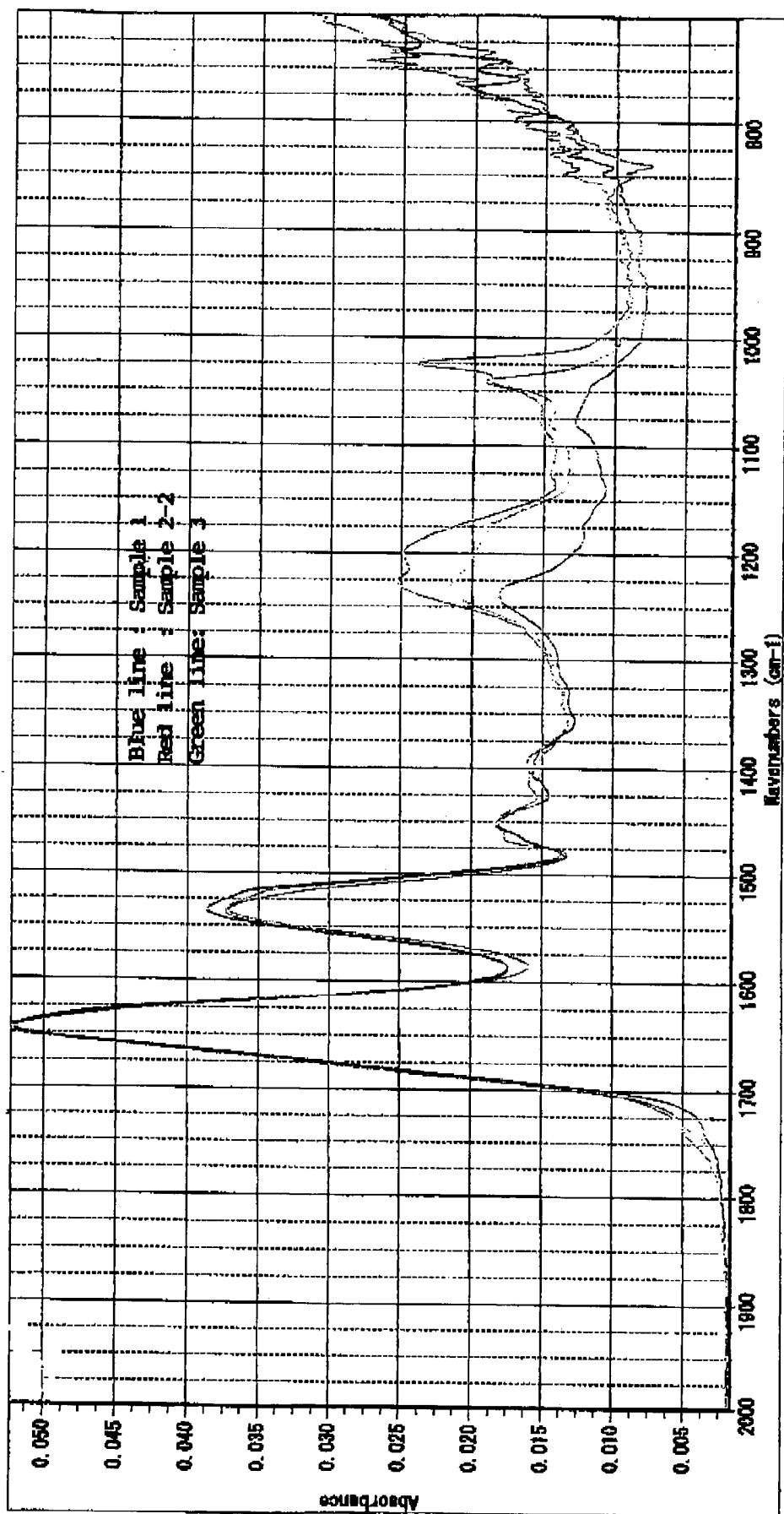


Fig. 4

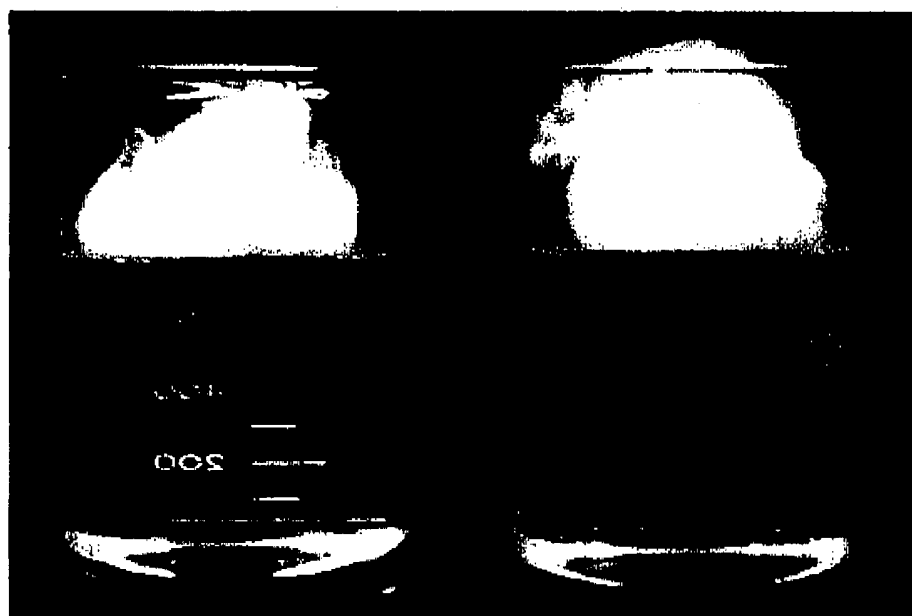
Sample 1  
(after 24hours)Sample 2-1  
(after 24hours)Sample 2-2  
(after 24hours)Sample 3  
(instantly)

Fig. 5(B)



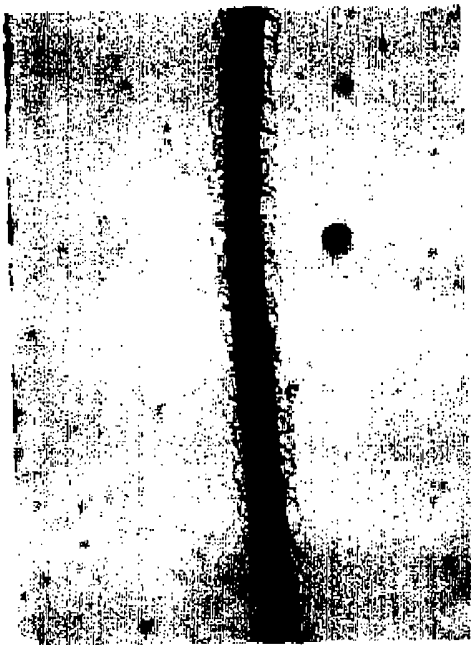
Sample 2-1

Fig. 5(D)



Sample 3

Fig. 5(A)



Sample 1

Fig. 5(C)



Sample 2-2